NUCLEAR MAGNETIC RESONANCE STUDY OF THE CRYSTALLIZATION OF SOME POLYMERS*

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A NUMBER of already classical methods, dilatometry, thermography and so on [1–4], are currently used to study the crystallization of polymers. In [5] one of us demonstrated that the method of nuclear magnetic resonance (NMR) can also be used in certain cases The present work aimed at a more detailed study of crystallization with this method.

EXPERIMENTAL

As the test specimens we used polyethyleneterephthalate (PETP) and the polyamides polyamide 6 (PA 6), polyamide 7 (PA 7) and polyamide 6-8 (PA 6-8) At room temperature these polymers may be in either the amorphous or crystalline state depending on their prior treatment. PETP was used as an amorphous, disoriented film. The starting specimens of the polyamides were crystalline To produce an amorphous structure they were melted in thin-walled sealed glass capillary tubes of 1–2 mm diam, and cooled in liquid nitrogen. For crystallization the amorphous specimens were heated to above their glass points The crystallinity was verified from X-ray diffraction patterns

The NMR spectra were taken as the derivative of the absorption signal from protons at 17 mc/s in the range 20–190° It took ~8 min to record one spectrum. Before this the specimens were held at the required temperature for 10–15 min

RESULTS AND DISCUSSION

The graphs given in [5] for the second moment ΔH_2^2 of the absorption line of an amorphous specimen of PETP as a function of the measuring temperature T indicate that crystallization processes occur between 100 and 120°. These are kinetic curves, i.e , they depend to some extent on t the time at which the specimen was held at the experimental temperature and the spectrum taken. But the crystallization process cannot be studied from these curves. To do this we found the second moment as a function of the holding time at given temperature (Fig. 1). At 80° ΔH_2^2 at first falls and than remains unchanged. At 90, 120 and 130°, judging from the graphs the second moment does not depend on the holding time. The spectra were taken not less than 20–30 min after the specimen had been put in the heating apparatus at given T. When PETP was held at 100 and 110° for a certain period there was an increase in the second moment.

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